#### (12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

# (19) World Intellectual Property Organization

International Bureau



# 

(43) International Publication Date 17 June 2004 (17.06.2004)

### (10) International Publication Number WO 2004/050786 A3

(51) International Patent Classification7:

C07C 67/00,

(21) International Application Number:

PCT/US2003/037411

(22) International Filing Date:

21 November 2003 (21.11.2003)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data: 60/429,630

27 November 2002 (27.11.2002)

- (71) Applicant (for all designated States except US): STEPAN COMPANY [US/US]; 22 West Frontage Road, Northfield, IL 60093 (US).
- (72) Inventors; and
- (75) Inventors/Applicants (for US only): HUTCHINSON, John, Christopher [US/US]; 6315 N. Wayne Ave. #3, Chicago, IL 60660 (US). LEE, Jungsik [US/US]; 2936 Greenleaf Ave., Wilmette, IL 60091 (US). FIGLEY, Timothy, M. [US/US]; 431 Brentwood Court, Roselle, IL 60172 (US). KRUEGER, Robert, A. [US/US]; 2512 N. Bosworth #3306, Chicago, IL 60614 (US).
- (74) Agent: WIMBISCUS, Thomas, J.; McAndrews, Held & Malloy, LTD., 34th Floor, 500 West Madison Street, Chicago, IL 60661 (US).

- (81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.
- (84) Designated States (regional): ARIPO patent (BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

#### Published:

- with international search report
- with amended claims
- (88) Date of publication of the international search report: 5 August 2004

Date of publication of the amended claims: 28 October 2004

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: METHOD OF MAKING METHYL ESTER SURFACTANTS

(57) Abstract: A method of making a methyl ester based surfactants is provided. The method includes the transesterification or esterification of a methyl ester feedstock and a poly(alkyl ether) feedstock.

PCT/US03/37411

## AMENDED CLAIMS

[Received by the International Bureau on 18 August 2004 (18.08.04): original claims 1-3 replaced by amended claims 1-24]

### AMENDED CLAIMS

[Received by the International Bureau on 18 August 2004 (18.08.04): original claims 1-3 replaced by amended claims 1-24]

Claim 1: (Amended) A method of making a methyl ester based surfactant comprising the steps of:

(a) providing a methyl ester feedstock comprising a methyl ester component having a general structure of the form R-C(O)OR', wherein R is an organic moiety and can be saturated or unsaturated, branched or linear, contain from about two to about twenty-four carbons, and wherein R' can be H or an organic moiety;

(b) providing a poly(alkyl ether) feedstock comprising a poly(alkyl ether) component having a general structure of the form HO(R''O)<sub>y</sub>R''', wherein R'' is an organic moiety, can be saturated or unsaturated, branched or linear, and containing from about one to about six carbons, wherein y ranges from about one to about 200, and wherein R''' is either H or an organic moiety that can be saturated or unsaturated, branched or linear, and contains from about one to about twelve carbons; and

(c) reacting the methyl ester feedstock with the poly(alkyl ether) feedstock...

Claim 2: (Canceled),

Claim 3: (Canceled).

Claim 4: (New): The method of claim 1, further comprising the step of providing a reaction catalyst.

Claim 5: (New): The method of claim 4, wherein the reaction catalyst is selected from the group consisting of acid catalysts, base catalysts or organo-metallic catalysts.

Claim 6: (New) The method of claim 4, wherein the reaction catalyst is selected from the group consisting of H<sub>2</sub>SO<sub>4</sub>, NaOCH<sub>3</sub>, and Sn(OH)C<sub>4</sub>H<sub>9</sub>.

Claim 7: (New) The method of claim 1, wherein the methyl ester feedstock has the following structural formula:

where x is 0 to 22.

Claim 8: (New) The method of claim 1, wherein the methyl ester feedstock is a methyl ester derived from a fatty acid having between 8 and 24 carbon atoms.

Claim 9: (New) The method of claim 1, wherein the methyl ester feedstock is selected from the group consisting of octanoic acid, decanoic acid, lauric acid, stearic acid, methyl octanoate, methyl decanate, methyl laurate, methyl stearate, and methyl palminate-oleate.

Claim 10: (New) The method of claim 1, wherein the poly(alkyl ether) feedstock has the following structural formula:

where y is from 1 to 200.

Claim 11: (New) The method of claim 1, wherein the poly(alkyl ether) feedstock is selected from the group consisting of poly(ethylene glycol) monomethyl ether, poly(ethylene oxide), poly(ethylene oxide) monomethyl ether, poly(propylene oxide), poly(propylene oxide) monomethyl ether, poly(ethylene oxide)-block-poly(propylene oxide), random copolymers of (ethylene oxide)-(propylene oxide), and mixtures thereof..

Claim 12: (New) The method of claim 1, wherein the reaction is carried out at a temperature between about 50°C and about 170°C.

Claim 13: (New) The method of claim 1, wherein the method further comprises the step of removing reaction-by-products as the reaction proceeds.

Claim 14: (New) A method of making a methyl ester based surfactant comprising:

- (a) providing a methyl ester feedstock comprising a fatty acid or fatty acid derived methyl ester;
  - (b) providing a poly(alkyl ether) feedstock; and
- (c) reacting the methyl ester feedstock and poly(alkyl ether) feedstock in the presence of a reaction catalyst.

Claim 15: (New) The method of claim 14, wherein the reaction catalyst is selected from the group consisting of acid catalysts, base catalysts or organo-metallic catalysts.

Claim 16: (New) The method of claim 14, wherein the catalyst is selected from the group consisting of H<sub>2</sub>SO<sub>4</sub>, NaOH<sub>3</sub>, and Sn(OH)C<sub>4</sub>H<sub>9</sub>.

Claim 17: (New) The method of claim 14, wherein the methyl ester feedstock is a methyl ester derived from a fatty acid having from about 2 to 22 carbon atoms.

Claim 18: (New) The method of claim 14, wherein the methyl ester feedstock is selected from the group consisting of octanoic acid, decanoic acid, lauric acid, stearic acid, methyl octanoate, methyl decanate, methyl laurate, methyl stearate, and methyl palminate-oleate.

Claim 19: (New) The method of claim 14, wherein the poly(alkyl ether) feedstock is selected from the group consisting of poly(ethylene glycol) monomethyl

ether, poly(ethylene oxide), poly(ethylene oxide) monomethyl ether, poly(propylene oxide), poly(propylene oxide) monomethyl ether, poly(ethylene oxide)-block-poly(propylene oxide), random copolymers of (ethylene oxide)-(propylene oxide), and mixtures thereof.

Claim 20: (New) The method of claim 14, wherein the method further comprises the step of removing reaction by-products as the reaction proceeds.

Claim 21: (New) A methyl ester based surfactant made according to the method of claim 1.

Claim 22: (New) The methyl ester based surfactant of claim 21, wherein the methyl ester based surfactant has the following structural formula:

where x ranges from about 0 to about 22, and y ranges from about 1 to about 200.

Claim 23: (New) The methyl ester based surfactant of claim 21, wherein x is between about 10 and about 20 carbon atoms.

Claim 24: (New) A methyl ester based surfactant made according to the process of claim 16.